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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/SE91/00454 (22) International Filing Date: 25 June 1991 (25.06.91) (30) Priority data: 9002276-5 28 June 1990 (28.06.90) SE (71) Applicant (for all designated States except US): ABB POW- DERMET AB [SE/SE]; Box 209, S-735 23 Surahammar (SE). (72) Inventors; and (75) Inventors/Applicants (for US only): EKBOM, Ragnar [SE/ SE]; Svedjevägen 8, S-612 00 Finspong (SE). SALWEN, Anders [SE/SE]; Alfhemsvägen 15, S-182 62 Djursholm (SE). (74) Agent: LARSSON, Håkan; ABB Corporate Research, Pa- tent Department, S-721 78 Västerås (SE).		(81) Designated States: AT (European patent), BE (European patent), CA, CH (European patent), DE (European pa- tent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (Euro- pean patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent), US. Published <i>With international search report.</i>
(54) Title: METHOD OF POWDERMETALLURGICALLY MANUFACTURING FULLY DENSE BODIES FROM HIGH TEMPERATURE MARTENSITIC Cr STEEL (57) Abstract A method of powdermetallurgically manufacturing a fully dense body of a high temperature martensitic chrome steel. A melt with an optimized composition (percentage by weight) of C 0.02 - 0.18 %; Si 0.05 - 0.5 % ; Mn 0.05 - 1.0 %; Cr 9.0 - 12.0 %; Ni 0.1 - 1.5 %; Mo 0.6 - 1.4 %; W 0.6 - 1.4 %; V 0.1 - 0.3 %; Nb 0.02 - 0.10 %; <u>N 0.02 - 0.18 %</u> ; B up to 0.01 %; Ti up to 0.1 %; Zr up to 0.1 %; Hf up to 0.2 %; Ta up to 0.2 %, the balance being Fe, and conventional impurities for this type of steel in conventional contents, is gas-atomized into a steel powder with a grain size of at most 1 mm. The contents of alloying elements included therein is adapted such that the molybdenum equivalent, expressed as ([%Mo] + 0.5[%W]), amounts to between 1.1 and 1.9 %, the total carbon and nitrogen content, ([%C] + [%N]), amounts to between 0.12 and 0.22 % and the total content of titanium, niobium, tantalum and hafnium, expressed as ([%Ti] + [%Nb] + 0.5[%Ta] + 0.5[%Hf]), amounts to at most 0.15 %. The powder is filled into a deformable container which is evacuated and essentially gas-tightly sealed before the container with the contained powder is consolidated into an essentially fully dense body by hot isostatic pressing or a combination of isostatic pressing and subsequent hot working.		

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Method of powdermetallurgically manufacturing fully dense
bodies from high temperature martensitic Cr steel

TECHNICAL FIELD

5 The invention relates to a method manufacturing fully dense bodies of a high temperature martensitic Cr steel, a number of material properties, preferably properties at elevated temperatures, being improved through a well balanced
10 composition and powdermetallurgical manufacture. The steel is manufactured by gas atomization into a steel powder, which is consolidated into bodies of essentially full density by hot isostatic pressing or a combination of isostatic pressing and subsequent hot working.

15 BACKGROUND ART

With rising oil prices considerable effort has been expended during the last few decades on achieving a more efficient
20 energy utilization. A measure with great effect is to increase the temperature in the energy conversion processes. This, in turn, requires new materials with improved high temperature properties.

25 One important material group are martensitic 9-12% Cr steels, which are attractive because of their high strength, low thermal expansion, good thermal conductivity, good ability to withstand stress corrosion and good damping properties.

30 Components of high temperature martensitic Cr steel with application within the energy field, for example in the generation of heat and electricity, are presently manufactured substantially with conventional ingot-based techniques.
35 One condition for a steel component of the stated type to receive the desired properties, primarily in connection with long-term use, that is, operating times of the order of magnitude of 100,000 hours or more in the temperature interval 550-630°C, is that the microstructure does not age

too rapidly. This, in turn, requires that the analysis is carefully balanced within certain intervals and that the material is given a good homogeneous composition. These requirements cannot be met with conventional methods such as ingot casting, ESR remelting, etc, especially for the materials mentioned, which contains elements which are prone to segregation, such as C, Si, E, Mo and W. The difficulties increase with the dimension of the casting.

- 10 Extensive development work has taken place, primarily within the scope of the conventional steel technique, to improve the alloying composition of this type of alloys. Alloys with compositions varying according to the following have been produced:

15

C	0.05 - 0.25%	Si	0.05 - 1.0%
Mn	0.05 - 2.0%	Cr	8 - 15%
Ni	0.1 - 2.0%	Mo	0.05 - 3.5%
W	up to 2.5%	V	0.1 - 0.5%
20 Nb	up to 0.5%	N	0.01 - 0.3%
E	up to 0.01%	Ti	up to 0.1%
Zr	up to 0.01%	Hf	up to 0.1%

- Ta up to 0.5%, the balance being Fe and conventional impurities for this type of steel in conventional contents. The
25 alloying elements included have been added based on the following.

- Carbon is added in order for martensite to form in the matrix and in order to form, together with Cr, Mo, W, Nb and
30 V and possibly Ti, Zr, Hf and Ta, carbides and/or carbonitrides.

Silicon is added as deoxidant.

- 35 Manganese is added preferably to bind oxygen and sulphur by forming MnS and MnO.

Chromium is added to obtain resistance to oxidation and wet corrosion and to form stable chromium carbides during tempering.

5 Nickel is added to suppress the formation of deltaferrite.

Molybdenum and tungsten are added to increase the strength by solution hardening and to increase the stability of the chromium carbides. In addition, molybdenum and tungsten
10 increase the corrosion resistance.

Vanadium is added to form a finely dispersed precipitation of carbonitride.

15 Niobium is added to form a finely dispersed precipitation of carbonitride.

Nitrogen is added to form stable carbonitride precipitations with vanadium and niobium.

20

Boron is added to increase the creep rupture strength.

Titanium, zirconium, hafnium and tantalum are added as supplement to or to partially replace Nb and V, that is, to
25 form finely dispersed carbonitride precipitations.

With the above materials, however, the desired improvements are not achieved, above all regarding properties such as impact strength, yield point, fatigue properties, creep
30 properties in the temperature range 500-600°C as well as stress corrosion properties in steel manufactured with an analysis according to the above.

SUMMARY OF THE INVENTION

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According to the present invention, an additional optimization is required of the composition relating to important alloying elements such as carbon, chromium, molybdenum.

tungsten, and boron, which according to the invention have been identified as key elements for which small changes in content are noticeably reflected in the properties of the material. In addition, important alloying elements such as carbon, chromium, molybdenum, tungsten and boron are greatly prone to segregation, which in connection with the solidification may result in the formation of residual melt regions, sometimes in the form of eutectics, with higher contents of the segregation-prone alloying materials, or in the occurrence of other serious defects such as residual porosity, residual ferrite, or cracks. Therefore, according to the invention, a steel powder is manufactured by gas atomization of the melt into a fine homogeneous and essentially segregation-free powder, which by subsequent hot-isostatic pressing or isostatic pressing followed by hot working is consolidated into an essentially dense body. In addition, by the careful inspection of the boron content and the avoidance of boron segregation, the tendency for cracking during a possible hot working, which normally occurs in conventionally manufactured material of this type as a consequence of boron segregation providing variations in the boron content within the body, is eliminated.

According to the invention, a fully dense body is manufactured from a high temperature martensitic chrome steel by means of powdermetallurgical methods, whereby a steel melt with the following optimized composition (percentage by weight)

30	C 0.02 - 0.18%	Si 0.05 - 0.5%
	Mn 0.05 - 1.0%	Cr 9.0 - 12.0%
	Ni 0.1 - 1.5%	Mo 0.6 - 1.4%
	W 0.6 - 1.4%	V 0.1 - 0.3%
	Nb 0.02 - 0.10%	N 0.02 - 0.18%
35	B up to 0.01%	Ti up to 0.1%
	Zr up to 0.1%	Hf up to 0.2%

Ta up to 0.2%, the balance being Fe, and conventional impurities for this type of steel in conventional contexts.

is atomized by means of gas atomization, into an essentially segregation-free steel powder with a powder particle size of at most 1 mm. The contents of certain alloying elements included are adapted such that the molybdenum equivalent, expressed as $([\%Mo] + 0.5[\%W])$, amounts to between 1.1 and 1.9%, that the total carbon and nitrogen content, $([\%C] + [\%N])$, amounts to between 0.12 and 0.22% and that the total content of titanium, niobium, tantalum and hafnium, expressed as $([\%Ti] + [\%Nb] + 0.5[\%Ta] + 0.5[\%Hf])$, amounts to at most 0.15%. The steel powder obtained is filled into a deformable container, which is evacuated and essentially gas-tightly sealed before the container containing the powder is consolidated into an essentially fully dense body by hot isostatic pressing or by a combination of isostatic pressing and subsequent hot working.

According to the optimization which the invention entails, the carbon content should be above 0.02% to provide the intended effect but below 0.18% in order for the carbide structure not to age too rapidly.

Nitrogen is added in contents exceeding 0.02% for the desired formation of carbonitrides but below 0.18% to avoid ageing by the growth of carbonitride precipitations.

According to the invention, the total carbon and nitrogen content, $([\%C] + [\%N])$, is adapted to between 0.12 and 0.22% to prevent carbonitrides and chromium carbides to coarsen too rapidly. In one embodiment of the invention, the total carbon and nitrogen content is adapted to be between 0.14 and 0.20%.

Silicon should be added in a content of at least 0.05% to provide the desired effect as deoxidant. However, the silicon content should not exceed 0.5% since silicon promotes the formation of deltaferrite and Laves phase.

The content of manganese should not be below 0.05% in order to bind oxygen and sulphur in the intended manner and not exceed 1.0% in order not to reduce the impact strength.

- 5 The chromium content should not be below 9.0% to provide the desired corrosion resistance, nor exceed 12.0% to avoid the formation of deltaferrite.

10 The nickel content should not be below 0.1% to suppress the formation of deltaferrite in the intended manner and not exceed 1.5% in order not to risk carbide coarsening, which is accelerated by nickel.

15 Molybdenum and tungsten are added in contents amounting to at least 0.6% each to raise the strength of the matrix by solution hardening and to increase the stability of the chromium carbides. In addition, molybdenum and tungsten contribute to an increase of the corrosion resistance. The content of either is not allowed to exceed 1.4%, in order
20 not to risk reduction of the creep strength and the ductility during long periods. According to the invention, the molybdenum equivalent, $([\%Mo] + 0.5[\%W])$, is adapted within an interval of between 1.1% and 1.9% to obtain optimum creep properties at current temperatures. According to one
25 embodiment of the invention, the molybdenum equivalent is adapted to amount to between 1.3 and 1.7%.

Boron is added to provide the desired increase of the creep rupture strength. In order not to deteriorate the ductility
30 at creep rupture or the weld properties, according to the invention the boron content is lower than 0.01% (100 ppm). In one embodiment of the invention, the boron content is adapted to between 0.0005% (5 ppm) and 0.0100% (100 ppm).

35 The vanadium content should not be below 0.1% to obtain the desired finely dispersed precipitation of carbonitrides and not exceed 0.3% in order not to provide too fast a coarsening of carbonitrides.

The niobium content should not be below 0.02% to obtain the desired finely dispersed precipitation of carbonitrides and not exceed 0.10% in order not to provide too fast a coarsening of carbonitrides.

5

The contents of titanium, zirconium, hafnium and tantalum, which are added as supplements to or partially to replace niobium and vanadium, that is, to form finely dispersed carbonitride precipitations, are limited according to the below:

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the content of Ti should not exceed 0.1%,
the content of Zr should not exceed 0.1%,
the content of Hf should not exceed 0.2%, and
15 the content of Ta should not exceed 0.2%

in order not to bind too much carbon and nitrogen. In addition, according to the invention, the total content of titanium, niobium, tantalum and hafnium, expressed as
20 $([\%Ti] + [\%Nb] + 0.5[\%Ta] + 0.5[\%Hf])$, is adapted to amount to at most 0.15%.

One condition for achieving the desired properties and keeping the close analyses defined according to the
25 invention is that the fully dense body is manufactured by gas-atomizing a steel melt of the specified composition into a homogeneous, essentially segregation-free steel powder with a grain size of at most 1 mm. This steel powder is filled into a deformable container which is evacuated and
30 essentially gas-tightly sealed before it and its contents of steel powder are consolidated by means of hot isostatic pressing or a combination of isostatic pressing and subsequent hot working, such as forging or extrusion, into an essentially fully dense body.

35

In one embodiment, the container containing the powder is consolidated by hot isostatic pressing, at a temperature of 1050-1200°C and a pressure of 75-150 MPa, into a density

exceeding 99% of the theoretical density of the steel.

Preferably, the container containing the powder is consolidated at a temperature of 1125-1175°C and a pressure of 90-110 MPa into a density exceeding 99.9% of the

5 theoretical density of the steel. Alternatively, the container containing the powder may be consolidated by cold isostatic pressing followed by hot working such as hot forging, hot extrusion or hot rolling into essentially full density.

10

The powder is preferably consolidated into components for use at elevated temperature within the energy region, such as turbine components, for example rotors, turbine discs, turbine blades and valves or components for steam and heat

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generation, for example pipes, pipe parts, tubes, valves, and steam collectors.

Since the powder, according to the invention, is rapidly solidified by means of gas atomization into a fine powder,

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the segregation problem is avoided and a homogeneous, essentially segregation-free steel powder is obtained. By adapting nozzle parameters, atomizing gas and other process parameters during the gas atomization, a powder with the desired grain size distribution is obtained. In the pro-

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duction of a segregation-free steel powder for manufacture of fully dense bodies by isostatic pressing, this means that the powder particle size is considerably below 1 mm.

By manufacturing the bodies by isostatic pressing, preferably by hot isostatic pressing, it is ensured that the residual porosity is very low, practically negligible, and that associated defects and deficiencies are essentially eliminated.

30

Components manufactured according to the invention are primarily intended to be used for a long time at elevated temperatures, preferably within the temperature interval 550-630°C. Therefore, after consolidation and any sub-

35

sequent working, the steel is usually tempered at temperatures within the interval 650-800°C.

5 It is self-evident that the manufacture of fully dense bodies of a martensitic 9-12% Cr steel according to the invention also comprises conventional measures taken during powdermetallurgical manufacture to ensure or check the quality, adjust dimensions, etc., with regard to the powder, the semi-manufactures, and/or the finished product.

10

CLAIMS

1. A method of powdermetallurgically manufacturing a fully dense body of a high temperature martensitic chrome steel,
5 **characterized in that** a melt with an optimized composition (percentage by weight) of

C	0.02 - 0.18%	Si	0.05 - 0.5%
Mn	0.05 - 1.0%	Cr	9.0 - 12.0%
10 Ni	0.1 - 1.5%	Mo	0.6 - 1.4%
W	0.6 - 1.4%	V	0.1 - 0.3%
Nb	0.02 - 0.10%	N	0.02 - 0.18%
B	up to 0.01%	Ti	up to 0.1%
Zr	up to 0.1%	Hf	up to 0.2%

- 15 Ta up to 0.2%, the balance being Fe, and conventional impurities for this type of steel in conventional contents, is gas-atomized into a steel powder with a grain size of at most 1 mm, wherein the contents of certain alloying elements included therein are adapted such that the molybdenum equivalent,
20 expressed as $([\%Mo] + 0.5[\%W])$, amounts to between 1.1 and 1.9%, the total carbon and nitrogen content, $([\%C] + [\%N])$, amounts to between 0.12 and 0.22%, and the total content of titanium, niobium, tantalum and hafnium, expressed as $([\%Ti] + [\%Nb] + 0.5[\%Ta] + 0.5[\%Hf])$, amounts to at
25 most 0.15%, whereupon said powder is filled into a deformable container which is evacuated and essentially gas-tightly sealed before the container with the contained powder is consolidated into an essentially fully dense body by hot isostatic pressing or a combination of isostatic
30 pressing and subsequent hot working.

2. A method according to claim 1, **characterized in that** the molybdenum equivalent is adapted to amount to between
35 1.3 and 1.7%.

3. A method according to claim 1 or claim 2, **characterized in that** the total carbon and nitrogen content is adapted to amount to between 0.14 and 0.20%.

4. A method according to claim 1, claim 2 or claim 3,
characterized in that the boron content is adapted to
amount to between 5 ppm and 100 ppm.

5 5. A method according to any of the preceding claims,
characterized in that the container with the contained
powder is consolidated into a body with a density exceeding
99% of the theoretical density of the steel by hot isostatic
pressing, at a temperature of 1050-1200°C and a pressure of
10 75-150 MPa.

6. A method according to any of the preceding claims,
characterized in that the powder is consolidated into
turbine components, for example rotors, turbine discs,
15 turbine blades and valves.

7. A method according to any of claim 1 to claim 5,
characterized in that the powder is consolidated into
components for steam and heat generation, for example pipes,
20 pipe parts, tubes, valves and steam collectors.

INTERNATIONAL SEARCH REPORT

International Application No PCT/SE 91/00454

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶ According to International Patent Classification (IPC) or to both National Classification and IPC IPC5: B 22 F 3/14, C 22 C 33/02, 38/22, 38/44														
II. FIELDS SEARCHED <div style="text-align: center;">Minimum Documentation Searched⁷</div> <table style="width: 100%; border: none;"> <tr> <td style="width: 25%; border: none;">Classification System</td> <td style="border: none;">Classification Symbols</td> </tr> <tr> <td style="border: 1px solid black; padding: 5px;">IPC5</td> <td style="border: 1px solid black; padding: 5px;">B 22 F; C 22 C</td> </tr> </table> <p style="text-align: center; font-size: small;">Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched⁸</p> <p>SE,DK,FI,NO classes as above</p>			Classification System	Classification Symbols	IPC5	B 22 F; C 22 C								
Classification System	Classification Symbols													
IPC5	B 22 F; C 22 C													
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹ <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 10%;">Category *</th> <th style="width: 70%;">Citation of Document,¹¹ with indication, where appropriate, of the relevant passages¹²</th> <th style="width: 20%;">Relevant to Claim No.¹³</th> </tr> </thead> <tbody> <tr> <td style="text-align: center; vertical-align: top;">Y</td> <td>Scandinavian Journal of Metallurgy, Vol. 11, 1982 S. Friberg: "HIP to Near Net Shape", see page 223 - page 225 --</td> <td style="text-align: center; vertical-align: top;">1-7</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">Y</td> <td>EP, A1, 0118702 (ASEA AB) 19 September 1984, see page 1, line 12 - line 19; page 4, line 32 - line 34; page 9, line 38 - page 10, line 14 --</td> <td style="text-align: center; vertical-align: top;">1-7</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">Y</td> <td>EP, A1, 0164678 (ALSTHOM-ATLANTIQUE) 18 December 1985, see page 1, line 1 - page 2, line 2; page 4, line 24 - line 31; page 5, line 25 - line 31; figure 1 --</td> <td style="text-align: center; vertical-align: top;">1-4</td> </tr> </tbody> </table>			Category *	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³	Y	Scandinavian Journal of Metallurgy, Vol. 11, 1982 S. Friberg: "HIP to Near Net Shape", see page 223 - page 225 --	1-7	Y	EP, A1, 0118702 (ASEA AB) 19 September 1984, see page 1, line 12 - line 19; page 4, line 32 - line 34; page 9, line 38 - page 10, line 14 --	1-7	Y	EP, A1, 0164678 (ALSTHOM-ATLANTIQUE) 18 December 1985, see page 1, line 1 - page 2, line 2; page 4, line 24 - line 31; page 5, line 25 - line 31; figure 1 --	1-4
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<div style="display: flex; justify-content: space-between;"> <div style="width: 48%;"> <p>* Special categories of cited documents:¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 48%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>														
IV. CERTIFICATION <table style="width: 100%; border: none;"> <tr> <td style="width: 50%; border: none;">Date of the Actual Completion of the International Search</td> <td style="width: 50%; border: none;">Date of Mailing of this International Search Report</td> </tr> <tr> <td style="border: 1px solid black; padding: 5px;">24th September 1991</td> <td style="border: 1px solid black; padding: 5px;">1991 -09- 3 0</td> </tr> <tr> <td style="border: none;">International Searching Authority</td> <td style="border: none;">Signature of Authorized Officer</td> </tr> <tr> <td style="border: 1px solid black; padding: 5px; text-align: center;">SWEDISH PATENT OFFICE</td> <td style="border: 1px solid black; padding: 5px;"> Nils Engnell </td> </tr> </table>			Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	24th September 1991	1991 -09- 3 0	International Searching Authority	Signature of Authorized Officer	SWEDISH PATENT OFFICE	 Nils Engnell				
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24th September 1991	1991 -09- 3 0													
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III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
Y	EP, A1, 0188995 (MITSUBISHI JUKOGYO KABUSHIKI KAISHA) 30 July 1986, see page 8, line 19 - page 10, line 1; page 31, line 1 - line 19 -- -----	1-4

ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.PCT/SE 91/00454

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the Swedish Patent Office EDP file on 91-08-30
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A1- 0118702	84-09-19	CA-A- 1226154	87-09-01
		JP-A- 59152272	84-08-30
		SE-B-C- 435272	84-09-17
		SE-A- 8300645	84-08-09
		US-A- 4568516	86-02-04
EP-A1- 0164678	85-12-18	FR-A-B- 2565251	85-12-06
		JP-A- 61000566	86-01-06
		US-A- 4689095	87-08-25
EP-A1- 0188995	86-07-30	JP-A- 61096062	86-05-14
		JP-A- 61096026	86-05-14